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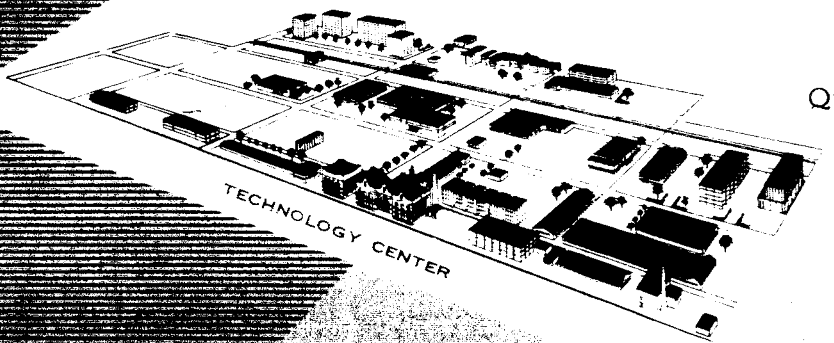

ARF 1220-QR 1
Quarterly Report Number 1

INVESTIGATION RELATING TO THE DEVELOPMENT OF CADMIUM TELLURIDE ENERGY CONVERTERS

National Aeronautics and Space Administration Headquarters
Office of Propulsion and Power Generation
Code RPP
Washington 25, D. C.

Contract No. NASw - 455

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Washington 25, D. C.

(Covering the Period from 10 July, 1962, to 10 October, 1962)

October 26, 1962

ABSTRACT

The objectives of the program are to construct experimental cadmium telluride (CdTe) single crystal solar cells and to measure their electrical properties which entails (A) producing suitable single crystal CdTe, (B) measuring its properties, (C) fabricating and evaluating CdTe solar cells, and (D) determining methods of optimum junction formation and depth, and of optimum electrical contacts. The controlled atmosphere furnace for zone refining, zone levelling, and production of single crystal CdTe was modified to insure greater temperature uniformity over the sample, narrower zone widths using baffled platinum zone heaters, and greater sensitivity to temperature changes. Boules and castings of CdTe were prepared to evaluate changes in the furnace. The major finding during this quarter was that essentially single crystal CdTe could be prepared in a cast layer form without seeding under conditions in which a boule of CdTe remains multicrystalline. Electrical properties of the single crystal cast CdTe are reported. Electroplating of silver is shown to lead to better contacts than the previously used chemical or evaporated techniques for p-type CdTe. In the analytical section, the A-value in the photovoltaic equation is discussed; the virtual inapplicability of the diffusion theory of p-n junctions for CdTe is shown even for nearly perfect junctions; calculations are made for a Shockley-Read recombination center in the space charge region of the junction; the importance of the junction region is stressed; and because the A-value is larger than unity, it is shown, under favorable conditions, that larger photovoltages are possible with

junction effects than one could obtain for a diffusion limited junction.

Future work is described.

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CADMIUM TELLURIDE ENERGY CONVERTERS

I. INTRODUCTION

This is the First Quarterly Report on an Investigation Relating to the Development of Cadmium Telluride Energy Converters for the National Aeronautics and Space Administration under Contract Number NASw-455, covering the period July 10, 1962, to October 10, 1962, inclusive. The nature of the program is to investigate the feasibility of utilizing cadmium telluride (CdTe) as a photovoltaic solar energy converter. The objectives of this contract are the construction of experimental cadmium telluride single crystal solar cells and the measurement of their electrical properties.

The work outline on this program is as follows: (A) Produce suitable crystal CdTe for fabricating solar cells; (B) Measure the physical and electrical properties of this base material; (C) Fabricate and evaluate experimental models of CdTe solar cells; and (D) Optimize the fabrication parameters, eg., optimum junction depth, junction formation, and electrical contacts.

Work during this first quarter was centered on parts (A) and (B), with the principal emphasis on part (A). Specifically, the major effort went into modifying the controlled atmosphere furnace which is used for zone refining, crystal growing, and zone levelling the CdTe, and preparing boules and castings of CdTe.

The major finding during this quarter is concerned with a cast layer of CdTe. Experimentally, a cast layer of CdTe which had been given a rapid ($\sim$ 0.5 cm/min) two pass zone refining treatment was found by x-ray analysis to be essentially single crystal, while a boule of CdTe in the same furnace which had been zone refined six times, at the same rate, showed a multi-crystalline structure quite consistent with the results of previous fast zone refining work. In addition, the cast layer had been grown between two smooth quartz surfaces and had highly polished surfaces, which raises the possibility of avoiding the multi-step lapping and polishing treatment that precedes junction formation by diffusion. X-ray, microscopic, and electrical properties (part B under work outline) of the cast CdTe are discussed.

After a description of the factual data, subsequent sections are devoted to analysis, the work accomplished in relation to the ultimate objectives, and finally, recommendations for future work.

II. SINGLE CRYSTAL PREPARATION

Cadmium telluride (CdTe) is a Group II - Group VI compound semiconductor with a band gap of 1.5 electron volts (ev).[1] Single crystals have been grown from both the vapor and the melt, however, the technology of preparing useful semiconductor material is not nearly as well developed for CdTe as it is for the better known semiconductors such as silicon or gallium arsenide.

Studies to date, here and elsewhere,[1,2] have shown that the electrical properties of CdTe are strongly dependent upon deviations from stoichiometry, and that the problem of obtaining useful semiconductor grade single crystals from the melt is intimately tied up with maintaining correct vapor pressures of the constituents. For example, past work using a simple gradient furnace with the polycrystalline CdTe in tapered quartz ampoules resulted in polycrystalline boules with numerous holes interspersed throughout, poor mechanical strength, large point-to-point variations in conductivity and also variations in conductivity type, that is n or p type.

In order to maintain a given vapor pressure of a constituent element and to allow equilibration between vapor, liquid and solid to take place in a reasonable time, a horizontal, controlled atmosphere furnace was constructed. Fundamentally, this furnace consists of three different resistive heating elements, providing, (1) a furnace for maintaining the boule at some temperature below the melting point, (2) another separate furnace for controlling the vapor pressure of a constituent, in particular, cadmium, and (3) a traversing zone refining heater. Representative temperatures for the furnace system during a zone refining experiment would be approximately 950°C for (1) above, 825°C for the cadmium heater, (2), and the zone heater, (3), should be hot enough to melt the CdTe. The zone heater has to provide a maximum value of 1090°C, the melting point, under stoichiometric conditions.[1,2]

The techniques for forming single crystal boules can be classified generally under the headings of (1) by seeding, or, (2) by non-seeding. In the non-seeding technique, a molten zone is allowed to traverse the material contained in a boat, such as a graphite or carbon black coated silica boat. Careful control of conditions is necessary in order to minimize bubbling; past experience in this laboratory indicates that the bubbling leads to non-uniform boules in both structure and electrical properties.

Important parameters [1] for growing single crystal CdTe are the choice of cadmium pressure, velocity of traverse of the molten zone, and the temperature gradient at the ends of the zone. Based on past work is this laboratory, it appears that uniform heating zones are necessary preconditions. To appreciate the significance of this idea, consider the situation during a given run. One has a furnace to maintain the solid CdTe boule at some given temperature in the region of 950-1000 C, then the function of the zone heater is to add enough heat to a small section such that it will reach the melting point, which will be close to 1090°c. If the maintaining furnace does not keep a nearly constant temperature over the boule, the zone width will fluctuate and hence the temperature gradient and the velocity of traverse of the molten zone will fluctuate. If the maintaining temperature is nearly constant, the controlling sensors for the zone heater can adjust the system in a short time. This minimizes the extent of the fluctuations in the temperature zone gradient, and the velocity of traverse of the liquid-solid interface.

Work during this period on the controlled atmosphere furnace and accessories was devoted to modifying the equipment in order to obtain (1) a more uniform temperature over the whole boule, (2) remaking the zone heater to provide a better control and a narrower zone. In part, this work dealt with the furnace itself, for example, new zone heaters were made, in which platinum wire replaced kanthal, and baffles were made to enclose the zone heater. This concentrated the heat onto a smaller segment of the boule. Zone widths have been reduced to less than a centimeter in contrast to five to seven centimeters utilized during early work on the controlled atmosphere furnace.

The importance of the narrow zone width can be appreciated in part by considering the problem of seeding in order to make single crystal boules. Since the seed crystals available are only in the centimeter or less size, it is necessary to keep from melting them entireley during the attempt to make a single crystal boule. Narrower zones are also useful for minimizing the waste material which is cut off after the zone refining treatment.

Returning to the problem of keeping uniform temperatures in the maintaining furnace, a number of modifications were made in the controlled atmosphere furnace insuring better uniformity. These include (a) making additional asymmetric windings to minimize gradient effects at the boundary, (b) making additional baffles, (c) utilizing different quartz and silica tubing diameters, and (d) lengthening the furnace. In addition, thermocouples were placed in closer proximity to the boule so that the response time of the overall furnace for temperature adjustments could be reduced.

Single Crystal Cast CdTe

During the modifications of the controlled atmosphere furnace, a monitoring technique was applied which consisted simply of preparing CdTe boules and evaluating them by x-ray analysis, optical microscopy and electrical probe measurements.

Recently, in an experiment on zone refining a polycrystalline boule some of the melted CdTe was inadvertently poured out of the boat after the fourth zone refining pass, providing a section of cast CdTe. In this experiment, the maintaining temperature over the boule was a little higher than usual, about 1020°C, and the zone width was wider. Two more passes were then made with the zone refining heater at the rate of approximately 0.5 cm/min, which was the same rate as for the previous four passes.

The resulting boule was multi-crystalline as has been found previously under these conditions. However, optical microscopic examination of pieces of cast CdTe showed a striking uniformity in their thermal etch pits. The surfaces of the cast material were quite smooth, so much so that interferometric studies of the slip lines could easily be made. Based on these observations, an estimate can be made that, aside from etch pit and slip regions, the surfaces were smooth to better than 400 Angstroms. This is roughly the limit of the technique. This smoothness, however, did not apply to the thick edges. The pieces overall had curved surfaces since they had been grown between the curved quartz tube surface and the smooth, curved silica boat containing the boule. Following the microscopic study, x-ray analyses were made using a Laue technique for both back reflection and transmission patterns. These studies showed that the cast CdTe was essentially single crystal. By "essentially" it is meant that the x-ray diagrams were definitely single crystal patterns, but that the individual Laue spots indicated a polygonization. It appears that by growing the single crystal on a curved surface, one obtains low angle boundary effects between different parallel portions of the single crystal. The apparent growth direction of the single crystal layers is about 15 degrees off the $\langle 111 \rangle$ crystallographic direction.

At the present, work is going on to prepare flat single crystal layers. Fortunately, thin layers allow more rapid equilibration of the solid and liquid with cadmium vapor pressure which will reduce preparation time. The difference between preparing single crystal boules and single crystal layers as a function of time of growth can be appreciated by comparing the passage rate of the zone heater. For example, deNobel used 0.5 to 0.8 cm/hr to reduce the bubble effect which militates, by distortion, against the growth of a single crystal material. The bubble effect is less pronounced for the layers apparently because the escape or release time is much shorter. The zone rate for preparing the single crystal layer was 0.5 cm/min or roughly fifty to sixty times faster than for the boule.

Attempts have already been made to grow thin layers of single crystal CdTe between two quartz plates. In addition, growth between a flattened side of the quartz reaction tube and a quartz plate has been tried. Results to date are negative. The difficulty, in large part, is that the CdTe is adhering to the quartz and tubing. This did not occur in the original casting experiment and it remains at present a problem. Currently an experiment is in progress on growing the layers in a shallow graphite boat with a flat graphite block for a weight just as a check on the concept of gradient zone freezing. The question, however, of sticking and non-sticking is being studied, and it may turn out crucial for this technique.

While work on preparing thin layers is in progress, the standard technique of growing a single crystal boule by seeding is also being considered. Seed crystals have been grown by sublimation in the furnace, and individual crystals suitable for seeding have been removed from the multicrystalline boules.

III. ELECTRICAL MEASUREMENTS

Past work in this laboratory on making electrical contact to n and p type CdTe showed that soldered indium makes an "ohmic" contact to n-type CdTe. This condition is based upon noise studies. For p-type CdTe, evaporated and chemically deposited silver and gold contacts were used. However, these contacts were not nearly as good as the indium on the n-type material. Work during this quarter in this area has led to an electroplating technique using silver deposition which leads to contacts having good mechanical strength and to which one can solder directly. To date these appear to be quite low resistance contacts, however, noise studies have not as yet been made, and hence the "ohmic" question is not settled.

Measurements made so far on the single crystal cast CdTe include resistivity, photoconductive decay lifetime, Hall coefficient, and Hall mobility. Apparatus is currently being assembled to combine photoconductive measurement with the PEM (photoelectromagnetic effect) in order to get the minority carrier lifetime.[3] Power density spectra from noise measurements may also be used if the minority carrier lifetime is not too small.

The p-type cast single crystal CdTe sample is shaped to have four self-probes by using a metallic brass mask and sand blasting. Originally silver electrodes were deposited and then the sample was shaped, but this procedure is being reversed because of the difficulty of sand blasting through the silver. The resistivity experiments were performed using currents ranging from 10 microamperes to 130 microamperes. Higher current values led to heating effects. The resistivity of the cast CdTe single crystal material is approximately 3000 ohm-cms.

The photoconductive decay experiments were conducted on an optical bench using a slotted wheel chopper rotated by a 10,000 rpm electrical motor with appropriate lens and focussing systems. The exponential voltage decay of the vacuum photocell was used to compare with the exponential decay of the sample. The voltage decays were observed on a Tektronix Type 545 oscilloscope using a Type K plug - in unit. The photoconductive decay lifetime of the CdTe is in the region of 25 microseconds. This is a majority carrier lifetime; that is, estimates of the change in conductivity under a known radiation intensity indicate that the observed decay time is a good approximation to the lifetime. Putting this another way, majority carrier trapping does not appear to be an important effect.

The Hall effect experiment was conducted with a dc electromagnet using six-inch pole pieces and a magnetic field intensity of 10,000 gauss; a current of 8.5 microamps was passed through the sample. The Hall voltage was determined from four voltage readings obtained by reversing the current and the magnetic field. the value of the Hall constant, R, was found to be 35.5×10^4 cm^3/coulomb.

The hole mobility by Hall effect was determined from the equation

$$R \rho^{-1} \frac{8}{3\pi} = \mu$$

leading to a value of 102 cm^2/volt sec for the hole mobility. From this value for the mobility and the resistivity, the majority carrier concentration was calculated to be 2×10^{13} cm^{-3}. Based upon the calculation of the np product for intrinsic CdTe with a band gap of 1.5 electron volts and effective masses for electrons and holes in the $0.1 m_o$ and $0.3\ m_o$ region respectively one can show that virtually no electrons are in the conduction band to carry current. Hence the approximation of a single type carrier is a good one.

This data can now be summarized and compared with other data for hole mobility.

TABLE I
Electrical Properties
Original
Single Crystal Cast Cadmium Telluride

Conductivity Type	p	
Resistivity	$\sim$ 3,000 ohm-cm	
Majority Carrier Lifetime	$\sim$ 25 microseconds	
Hall Constant, R	35.5 x 10^4 cm^3/coulomb	
Hall effect, hole mobility (room temp.)	$\sim$ 102 cm^2/volt-sec	65 cm^2/volt-sec* 35 cm^2/volt-sec**
Carrier concentration (room temp.)	2 x 10^{13} cm^{-3}	

* D. de Nobel, Philips Res Rpts 14, pp 430-492, 1959

** This value is taken for approximately the same carrier concentration from Kroger and de Nobel, J. Electronics 1, 190, 1955

IV. ANALYSIS AND THE RELATION OF THE DATA TO THE ULTIMATE OBJECTIVES

The theory of p - n junction photovoltaic cells has been[4, 5, 6, 7, 8, 9] discussed by a number of authors, and, in particular, it has been noted[6, 8, 10] that the empirical equation determined from measurements is not in agreement with the simple diffusion theory of p - n junction.[11] For example, the equations for the observed photocurrent and open circuit voltage (under the assumptions of negligible internal resistance, and infinite impedence for the shunting resistance) are given by

$$I = I_s - I_o(e^{eV/AkT} - 1); \quad \left(\frac{I_s}{I_o} + 1\right) = e^{\frac{eV_{oc}}{AkT}}$$

where I_s is the short circuit current

I_o is the reverse saturation current

V is the voltage change in the barrier height under illimination

V_{oc} is the open circuit photovoltage

It is the parameter A, however, that makes this an empirical equation. Only under the condition that A is unity does this equation reduce to the one derived from the diffusion theory of p - n junctions. A values, depending on the cell, can get as high as 3, however they usually appear to lie between 2 and 3 for silicon cells. It is the intention in this section to discuss briefly the effect of these results on pn junction CdTe photovoltaic cells.

Let us assume that the simple diffusion theory for p - n junctions did apply so that A = 1 and I_o could be estimated from the diffusion current approximation. Assume 3 ohm-cm material, and for simplicity let us consider only the contribution due to the electrons as minority carriers on the p-side of the junction. From the np product approximation

$$np = N_c N_v e^{-E_g/kt}$$
$$= 3.8 \times 10^{36} e^{-1.5/0.026} \qquad \text{(room temperature)}$$

using 0.1 m_o as the effective mass for electrons; 0.3 m_o as the effective mass for holes, and a band gap of 1.5 electron volts so that

$$np \cong 3 \times 10^{11}$$

Now, for 3 ohm-cm material, p-type material, one has

$$p \cong 2 \times 10^{16}$$

and hence

$$n \cong 1.5 \times 10^{-5}.$$

Now for the number of electrons which diffuse over the junction per second, one considers the thermal generation rate, G, and assumes the random motion of the electrons averages out to the approximation that all carriers generated within a diffusion length, L, of the junction will go over. Therefore, the current due to the electrons is

$$I_{oe} = (J_o A)_e = (eG\ L\ A)_e$$

and since under equilibrium conditions the generation rate equals the recombination rate, $R = n/\tau$ (where a constant minority lifetime is assumed), then

$$(J_oA)_e = e\frac{n}{\mathcal{T}}(D\mathcal{T})^{1/2}$$

$$= e\,n\left(\frac{D}{\mathcal{T}}\right)^{1/2} A$$

Assuming further that tau, $\mathcal{T}$, is 10^{-8} seconds, and using D for electrons of about 18 cm^2/sec at room temperature, then

$$(J_oA)_e = 1.6 \times 10^{-19} \times 1.5 \times 10^{-5} \times (18 \times 10^{8})^{1/2} \text{ A}$$

$$(J_oA)_e \cong 1.1 \times 10^{-19} \text{ A amperes}$$

or less than one electron per second per centimeter squared.

With such extremely small diffusion currents even for minority carrier lifetimes in the 10^{-8} seconds regions, it is quite certain that the generation and migration of carriers in the space charge region of the junction will dominate the diffusion current even for a nearly perfect junction. In part, then, this brief calculation serves to emphasize the importance of the junction itself and that A values in the photovoltaic equation for CdTe would be expected to be junction dependent, as is the case apparently for silicon.

As the next level of approximation, assume the existence of recombination centers near the middle of the band gap - Shockley-Read model - and let us calculate, following Sah et al[12], an approximate value for I_o with a symmetric junction having the parameters listed previously: 3 ohm-cm material and a minority carrier lifetime of

10^{-8} seconds. Further, for simplicity, assume an abrupt junction; roughly, then, one gets a junction width of about 2000 angstroms. The generation rate in the junction with this model is

$$G_j = \frac{n_i}{2\mathcal{T}}$$

where n_i is the intrinsic concentration $\sim 5.5 \times 10^{5}$ at room temperature and $\mathcal{T}$ is 10^{-8} seconds. Combining this with the junction width of 2000 angstroms, one has

$$(I_o)_i = (J_o A)_j = e \frac{n_i}{2\mathcal{T}} \cdot W \cdot A \cdot$$

$$= \frac{e \cdot 5.5 \times 10^{5} \times 2 \times 10^{-5} \quad A}{2 \times 10^{-8}} \quad \text{amperes}$$

$$= 1.6 \times 10^{-19} \times 5.5 \times 10^{-8} \quad A$$

$$= 8.8 \times 10^{-11} \quad A$$

or approximately 10^{-10} A amperes.

It is true, of course, that junction widths calculated for silicon solar cells are smaller by a factor of ten,due to higher doping, than the one in this example, but this order of magnitude calculation does serve to illustrate the importance of effects within the junction. This model leads to an A value of 2, which is still smaller than the values usually obtained in a silicon solar cell.

Referring to the A values again, it is interesting to note that generation within the junction need not be deleterious to the overall open circuit voltage as compared with only diffusion current. In fact, because the A values are larger than unity, larger resultant photovoltages are possible. For example, if, for a CdTe cell, one uses a short circuit current, $I_s = 10^{-3}$ A, and assumes a minority carrier lifetime of 10^{-7} seconds rather than 10^{-8} and using

$$\left(\frac{I_s}{I_o} + 1 \right) = e^{eV_{oc}/AkT}$$

where A equals unity for diffusion theory, A = 2 for the Shockley-Read model and the I_o are calculated as before (except that $(J_oA)_e$ is doubled to get the diffusion current for both sides of the junction) one finds $V_{oc} = 0.91$ for the diffusion theory case, and $V_{oc} = 0.96$ for the junction generation-recombination case.

Since, therefore, space charge effects in the junction are undoubtedly going to be very important, it should be realized that this by itself does not necessarily preclude one from obtaining a useful photovoltaic converter. However, this result was obtained by assuming a minority carrier lifetime of 10^{-7} seconds, and a good junction.

The reason for carrying out these simple analyses is to explain the emphasis, during the first quarter, on the bulk material. While it is true that the fabrication of junctions tends to reduce the minority carrier lifetime, one should at least use bulk material with minority carrier lifetimes larger than some minimum acceptable value. Preliminary results of past work indicate that CdTe will have lower fabrication temperatures than for silicon, and hence, it is hoped, less fabrication damage. Good starting material, therefore, may not be drastically changed in so far as lifetime is concerned.

Previous work on the absorption edge of CdTe shows it to be sloping, that is, much more like silicon than gallium arsenide, and hence minority carrier contributions on both sides of the junction are expected. It follows, also, that to get a good converter for white radiation, that is, good overall collection efficiency, one wants long diffusion lengths. Since the diffusion constants are smaller than silicon, good minority lifetimes for both carriers are essential.

Second, effects in the junction for CdTe will probably be more important than for silicon. For example, a calculation of I_o for silicon junction, using 3 ohm-cm material, 10^{-8} second minority carrier lifetimes, and the Shockley-Read model leads to currents in the 10^{-6} ampere region as contrasted with 10^{-10} seconds for CdTe. Small currents, therefore, through some involved paths in the junction would appear to effect CdTe more than silicon. Finally, it is the contention of this report that the more complicated effects in the junction will have to be considered even more seriously than in silicon because it is a larger band gap material with the possibility of larger internal fields and greater sensitivity to defects.

Work during this period has been concerned with the base material; (A) preparing single crystals, (B) measuring certain electrical properties of it, and (C) making contacts to this p-type material. The previous discussion covered the reasons for work on (A); and since the material needs to be evaluated, this covers (B). Specifically, the work on single crystal cast layers holds out the hope of more rapid growth of single crystals in which the zone levelling and crystal growth are carried out in one operation. This is possible by using the sensitivity of the conductivity to the pressure of cadmium over the sample during growth. It is interesting to note that the mobility value obtained on the cast material is higher than the literature values, and one measure of crystal perfection often used is that of mobility. However, this mobility figure should be considered only as a preliminary value. Assuming it were true, though, this would indicate better CdTe than that used for the values published in the literature.

The problem of contacts can be appreciated by considering the general descriptive equation for the photocurrent used by Prince and Wolf[13]:

$$I = I_s - I_o \left(e^{\frac{e(V + IR_s)}{AkT}} - 1 \right) - \frac{V}{R_{sh}}$$

where R_s is the internal resistance and R_{sh} is the shunt resistance.

As is evident, this more general equation takes into account internal resistance and shunting which were previously neglected. The internal resistance covers not only the semiconductor itself, but includes contact resistance. It is obvious from the equation that one wants to minimize R_s as much as possible consistent with other requirements such as collection efficiency. But as far as contact resistance is concerned, one would hope to eliminate it. The experiments on electroplating of silver to the p-type CdTe single crystals are directed along these lines. To date, one can say that the contacts are better than the previous chemical and evaporated contacts to p-type material used in this laboratory.

V. FUTURE WORK

Single crystal growing of CdTe will continue. This covers not only the experiments on the single crystal layers, but also the seeding of zone refined multicrystalline boules. Electrical measurements will include resistivity, Hall constant, Hall mobility, photoconductivity lifetime, and it is hoped, minority carrier lifetime using the combination of the PEM and the photoconductivity effects.

p - n junctions will be formed on the p-type material by diffusing in a thin evaporated layer of a donor, specifically indium. These experiments are in progress. The diodes formed will be studied for open circuit photovoltage, short circuit current and the I - V curve will be traced. For comparison purposes, the diodes will be studied under small forward bias and compared with the general equations. A low angle grinding device has been prepared, and it is hoped that useful information of the depth of the junction will be obtained. Combining this information with the optical absorption characteristics, it is hoped that data will be obtained on the degradation (if any) of the minority carrier lifetime during fabrication of the junction. The data will then be analyzed and reported in the next quarterly report.

VI. LOGBOOKS AND CONTRIBUTING PERSONNEL

All data presented in this report are contained in ARF Logbooks numbers 12753, 12754, 12755. Personnel who have contributed to this work and the preparation of this report are O. Brandt, A. van den Heuvel, M. Scott, and R. J. Robinson.

Respectfully submitted,

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